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(54) Title: INSECTICIDAL SEED COATING (57) Abstract The present invention relates to an insecticidal coating for a seed comprising one or more binders selected from the group consisting of polymers and copolymers of polyvinyl acetate, methyl cellulose, polyvinyl alcohol, vinylidene chloride, acrylic, cellulose, polyvinylpyrrolidone and polysaccharide and an insecticide and filler wherein the binder forms a matrix for the insecticide and filler resulting in a substantially non-phytotoxic seed coating; methods for producing said coating and the seed treated with the above described coating. Additionally the invention concerns a method of controlling soil-borne insect pests, particularly from the order of Coleoptera, such as Diabrotica spp., comprising applying to a seed an insecticidal coating as described above.		

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INSECTICIDAL SEED COATING

BACKGROUND OF THE INVENTION

5 Numerous technologies are currently utilized to treat seeds with the desired purpose of enhancing crop performance. These treatments include *inter alia* the coating, pelleting and/or film overcoating of seeds. A range of fungicide treatments as dusts, liquids and slurries have long been used to control soil and seed-borne diseases in vegetable seeds. However, the use of insecticides for vegetable seed treatment has lagged behind that of fungicide seed
10 treatments, and moreover, seed treatment of agronomic crops is even less advanced than that for vegetable seed.

Recent developments in seed treatment technology have focused on the use of techniques to deliver pesticides to seeds. Film-coating has been studied as a means of
15 delivering insecticides such as benfuracarb, chlorpyrifos, chlorfenvinphos and others. In the past, the concentration of these insecticides and other active ingredients present in the seed coating was limited due to direct phytotoxic effect of the insecticide on the seed. Using the instant seed coating of the present invention solves the problem of direct insecticide phytotoxicity to the seed.

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SUMMARY OF THE INVENTION

The present invention relates to an insecticidal coating for a seed comprising one or more binders selected from the group consisting of polymers and copolymers of polyvinyl
25 acetate, methyl cellulose, polyvinyl alcohol, vinylidene chloride, acrylic, cellulose, polyvinylpyrrolidone and polysaccharide, one or more filler(s), and an insecticidally effective amount of an insecticide, preferably an organophosphate, phenyl pyrazole, pyrethoid, or imidacloprid insecticide wherein the binder forms a matrix for the insecticide and filler resulting in a substantially non-phytotoxic seed coating.

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In a preferred embodiment, the invention concerns an insecticidal coating for a corn seed comprising a binder in an amount from about 0.01 to about 15% of the weight of the seed wherein said binder is a vinyl acetate-ethylene copolymer or polymer or copolymer of

vinylidene chloride, a filler in an amount of up to about 70% of the weight of the seed, an insecticide in an amount from about 0.005 to about 50% of the weight of the seed and selected from the group consisting of imidacloprid, terbufos, chlorpyrifos, fipronil, tefluthrin, chloroethoxyfos, tebupirimfos, and mixtures thereof, wherein said binder forms a matrix for the insecticide and filler(s) resulting in a substantially non-phytotoxic seed coating.

In another preferred embodiment the invention relates to a method of controlling soil-borne insect pests, such as corn root worm, comprising applying to a seed an insecticidal coating as described above wherein said insecticide is applied to the seed in an insecticidally effective amount to control insect pests.

Additionally the invention concerns a method of preparing a coated seed comprising a) mixing one or more binders with an insecticide selected from the group consisting of imidacloprid, terbufos, chlorpyrifos, tefluthrin, fipronil, chloroethoxyfos, tebupirimfos, and mixtures thereof, wherein the binder serves as a matrix for the insecticide; b) applying the mixture to a seed; c) allowing the mixture to dry on said seed and d) applying a film overcoating to the seed.

In still another embodiment the invention is concerned with a method of protecting emerging seedlings of a crop plant from one or more insect organisms in the seed growing soil environment which comprises applying to the seeds of the crop plant an insecticidally effective amount of an insecticidal coating as described herein above.

The seed growing environment as used herein includes that area directly surrounding the seed and young roots.

Frequently when pesticides are applied to seeds, the pesticide may cause physiological damage to the seed or seedling due to phytotoxic effects of the concentrated pesticide in or around the seed. The present invention is novel in that pesticides may be applied in a polymer matrix at pesticidally effective concentrations without having an adverse phytotoxic effect on the emerging seedling. In particular it has been found that the use of the coating of the present invention is effective in the control of insect pests, such as Coleoptera and Lepidoptera insects.

It is a most important object of the present invention to provide a seed coated with a coating which protects the seed or the emerging seedling from physiological damage potentially caused by the insecticidal ingredient of the coating.

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A still further object of the invention is to provide a hybrid corn seed coated with an insecticide which aids in the control of Coleopteran pests, such as Diabrotica insects, and particularly corn rootworm larvae.

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Another object of the invention is to provide a seed which is coated with an insecticide whereby the use of the insecticide in the coating increases the safety of using said insecticide by reducing operator exposure.

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Still another object of the invention is to provide an insecticidal seed coating wherein the insecticide is encapsulated within a matrix that provides for the controlled release of the insecticide over a prolonged period of time.

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Yet another aim of the invention is to improve the flowability of seed through a seed planter which will ultimately result in more uniform seed planting.

DETAILED DESCRIPTION OF THE INVENTION

The present invention relates to improved seed coatings which are superior to conventional seed coatings comprising insecticidal components. The invention also relates to the use of insecticides in seed coatings wherein the insecticide has heretofore been ineffective against certain insect pests, however, may be formulated in the seed coating of the present invention in sufficiently high amounts to be effective against those same insect pests without causing unacceptable phytotoxicity to the seed.

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As used herein the term coated seed means a seed that has been subjected to a procedure whereby the seed is treated with one or more adhering coating layers.

The coating described herein includes two main components: a) one or more binders and b) an insecticide. The binder serves as a matrix for the insecticide and is preferably present in the seed coating in an amount sufficient to prevent or reduce the levels of phytotoxicity caused by the insecticide.

5

The binder component of the coating is composed preferably of an adhesive polymer that may be natural or synthetic and is without phytotoxic effect on the seed to be coated. The binder may be selected from polyvinyl acetates, polyvinyl acetate copolymers (-ethylene), polyvinyl alcohols, polyvinyl alcohol copolymers, celluloses, including ethylcelluloses and
10 methylcelluloses, hydroxymethylcelluloses, hydroxypropylcellulose, hydroxymethylpropylcelluloses, polyvinylpyrrolidones, dextrans, maltodextrins, polysaccharides, fats, oils, proteins, gum arabics, shellacs, vinylidene chloride, vinylidene chloride copolymers, calcium lignosulfonates, acrylic copolymers, starches, polyvinylacrylates, zeins, gelatin, carboxymethylcellulose, chitosan, polyethylene oxide, acrylimide polymers and copolymers,
15 polyhydroxyethyl acrylate, methylacrylimide monomers, alginate, ethylcellulose, polychloroprene and syrups or mixtures thereof. Preferred binders include polymers and copolymers of vinyl acetate, methyl cellulose, polyvinyl alcohol, vinylidene chloride, acrylic, cellulose, polyvinylpyrrolidone and polysaccharide. The above-identified polymers include those known in the art and for example some are identified as Rhoplex™ B-60A, Methocel™
20 A15LV, Methocel™ E15LV, Cellosize™ QP, AirFlex™ 500, Daratak™ SP 1090, Elvanol™ 85-30, Rhoplex™ AC-33-NP, Rhoplex™ B-85 and Vinamul™ 18132. Particularly preferred classes of polymers include polymers and copolymers of vinylidene chloride and vinyl acetate-ethylene copolymers.

25 The amount of binder in the coating will be in the range of about 0.01 to 15% of the weight of the seed. A preferred range will be about 0.1 to 10.0 % of the weight of the seed.

The binder must be chosen so that it serves as a matrix for the insecticide. While the binders disclosed above may all be useful as a matrix, the specific binder will depend on the
30 properties of the insecticide. The term "matrix" as used herein means a continuous solid phase of one or more binder compounds and contains vacancies, voids or spaces occupied by the insecticide and filler. The term matrix is given a broad meaning and includes what may be viewed as a matrix system, a reservoir system or a microencapsulated system. In general a

matrix system consists of an insecticide and filler uniformly dispersed within a polymer, while a reservoir system consists of a separate insecticide phase, insecticide particles or droplets physically dispersed within a surrounding, rate limiting polymeric phase. Microencapsulation includes the coating of small particles or droplets of liquids. The term microencapsulation has not only been applied to coated particles or droplets but also to dispersions in a solid matrix. Without being limited to the specific encapsulating system (matrix, reservoir or microencapsulated) the term matrix is meant to be inclusive of the above listed systems.

The invention further contemplates the use of fillers, such as absorbent or inert fillers, in the insecticidal coating. It has been discovered that the use of fillers in the coating is particularly effective for protecting the seed during stress conditions. Fillers for such formulations are known in the art and may include woodflours, clays, activated carbon, sugars, diatomaceous earth, cereal flours, fine-grain inorganic solids, calcium carbonate and the like. Clays and inorganic solids which may be used include calcium bentonite, kaolin, china clay, talc, perlite, mica, vermiculite, silicas, quartz powder, montmorillonite and mixtures thereof. Sugars which may be used include dextrin and maltodextrin. Cereal flours include: wheat flour, oat flour and barley flour. Preferred fillers include diatomaceous earth, perlite, silica and calcium carbonates and mixtures thereof. For example, a product containing diatomaceous earth and amorphous silica such as that manufactured by Celite Corporation (Celite™) is most preferred. One skilled in the art will appreciate that this is a non-exhaustive list of materials and that other recognized filler materials may be used depending on the seed to be coated and the insecticide used in the coating.

The filler is chosen so that it will provide a proper microclimate for the seed, for example the filler is used to increase the loading rate of the active ingredient and to adjust the control-release of the active ingredient. A filler aids in the production or process of coating the seed. The effect varies, because in some instances formulated insecticidal compounds will comprise a filler. The amount of filler used may vary, but generally the weight of the filler components will be in the range of about 0.005 to 70% of the seed weight, more preferably about 0.01 to 50% and most preferably about 0.1 to 15%. The filler may be supplied in the coating of the invention with the insecticidal component. The specific examples as described herein utilize readily available commercial formulations of known insecticides wherein filler material is included in the formulation.

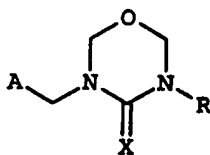
Suitable insecticides include those selected from azoles, for example, triazoles, azin^{es}, pyrethroids, organophosphates, caramoyloximes, pyrroles, pyrazoles, pyridines, amidines, halogenated hydrocarbons, and carbamates and combinations and derivatives thereof. Particularly suitable classes of insecticides include insect growth regulators (e.g., MimicTM)

5 organophosphates (e.g., FortressTM), phenylpyrazoles and pyrethroids. Preferred insecticides are those known as terbufos, chlorpyrifos, fipronil (RegentTM), chlorethoxyfos, tefluthrin, fiproles, phenoxycarb, diofenolan (AwareTM), pymetrozine (ChessTM), carbofuran, tebupirimfos, and imidacloprid (GauchoTM, ConfidorTM, AdmireTM), including imidacloprid analogs, such as

10 (substituted or unsubstituted) nitro-, oxo-, or cyano-substituted-guanidines, enamines, iminomorpholines, piperazines, iminopiperazines, oxapiperazines, oxadiazines, oxapyridines, diazocyclohexanes, diazolidines, and morpholines. Most preferred are imidacloprid and imidacloprid analogs, for example, those disclosed in U.S. Pat. No. 5,034,524, terbufos, chlorpyrifos, fipronil, tefluthrin, chloroethoxyfos, tebupirimfos, and mixtures thereof, for example, imidacloprid and tefluthrin.

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It has been discovered that the seed treatment of this invention demonstrates particular applicability to compounds of the general formula (I):



(I),

20 in which

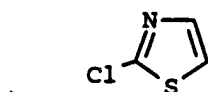
A is an unsubstituted or mono- to tetrasubstituted, aromatic or non-aromatic, monocyclic or bicyclic heterocyclic radical, where one to two of the substituents of A can be selected from the group consisting of halo-C₁-C₃alkyl, cyclopropyl, halocyclopropyl, C₂-C₃alkenyl, C₂-C₃alkynyl, halo-C₂-C₃alkenyl, halo-C₂-C₃alkynyl, halo-C₁-C₃alkoxy, C₁-C₃alkylthio, halo-C₁-C₃alkylthio,

25 allyloxy, propargyloxy, allythio, propargylthio, haloallyloxy, haloallylthio, cyano and nitro, and one to four of the substituents of A can be selected from the group consisting of C₁-C₃alkyl, C₁-C₃alkoxy and halogen;

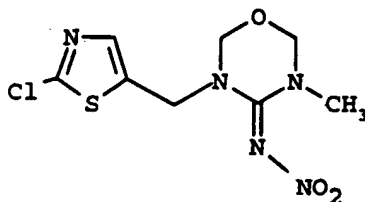
R is hydrogen, C₁-C₆alkyl, phenyl-C₁-C₄alkyl, C₃-C₆cycloalkyl, C₂-C₆alkenyl or C₂-C₆alkynyl; and X is N-NO₂ or N-CN,

30 or, if appropriate, a tautomer thereof, in each case in free form or in salt form.

An especially preferred insecticide of the general formula (I) is the compound wherein A



and X is N-NO₂ which yields the compound of formula II:



(II)

The compound of formula II may be used alone or in combination with other pesticides, for example, with tefluthrin. Other combinations of pesticides, for example, pyrethroids and organophosphates, are also suitable.

Also included are insect growth regulators for example, methoprene and hydroprene. These are well known to those skilled in the art.

The amount of the insecticide in the coating will vary depending on the type of seed and particular active ingredient, but in general will range from about 0.005 to 50% of the weight of the seed. A preferred percent range for the insecticide is about 0.01 to 40%. A most preferred percentage range is about 0.05 to 20.0%. In particular, the range for chlorpyrifos will be about 0.5 to 20.0%, a preferred range will be about 1.0 to 15%; the range for tefluthrin will be about 0.01 to 10.0%, a preferred range will be about 0.05 to 5.0%; the range for tebupirimfos will be about 0.01 to 5.0%, a preferred range from about 0.05 to 3.0%; the range for fipronil will be about 0.01 to 10.0%, and a preferred range will be about 0.05 to 5.0%; and the range for imidacloprid and imidacloprid analog insecticides will be about 0.01 to 10%, and a preferred range will be about 0.05 to 5%.

As one skilled in the art can appreciate the exact amount will vary depending of the size of the seed to be coated. The insecticide of the coating must not inhibit germination of the seed and should be efficacious during that time in the target insects life cycle which causes injury to a crop plant. One skilled in the art will appreciate that this time will vary depending on the target insect among other factors. In general the coating will be efficacious for approximately 0 to 120 days after sowing. The coating of the present invention will contain an amount of insecticide that is insecticidally effective. An insecticidally effective amount as used herein means that amount of insecticide that will kill insect pests in the larvae or pupal stage of growth or will consistently reduce or retard the amount of damage produced by insect pests.

10

When the insecticidal component used for the coating is an oily type formulation and little or no filler is present, it may be necessary to hasten the drying process by drying the formulation. This optional procedure may be accomplished by means well known to those skilled in the art and includes the addition of calcium carbonate, kaolin or bentonite clay, perlite, diatomaceous earth or any adsorbent material added preferably concurrently with the insecticidal coating layer to absorb the oil or excess moisture. The amount of calcium carbonate or related compounds to dry charge the solution will be in the range of about 0.5 to 10.0 % (w/w).

15

The coatings formed by the invention are capable of effecting a slow rate of release of the insecticide by diffusion or movement through the matrix to the surrounding medium.

20

Virtually any crop seed can be treated in accordance with the invention, such as cereals, vegetables, ornamentals, and fruits. Particular crop seeds are selected from the group of corn (sweet and field), soybean, wheat, barley, oats, rice, cotton, sunflower, alfalfa, sorghum, rapeseed, sugarbeet, Brassica spp., tomato, bean, carrot, tobacco and flower seed, for example, pansy, impatiens, petunia and geranium. The most preferred seeds include corn and soybean.

25

Preferred target pest organisms include soil borne insects of the order.

30

Coleoptera particularly *Diabrotica* sp. (Western, Southern and Northern corn rootworm), for example, *D. balteata*, *D. virgifera*, *D. undecimpunctata*, and *D. longicornis*; *Melanotus* sp. (Corn wireworm); *Phyllophaga* sp. (White grubs, Wireworms, and False wireworms); *Limonius* sp. (Sugarbeet wireworms) and *Agrotis* sp. (Wheat wireworms, White grubs and Seed
5 maggots):

Lepidoptera particularly *Peridroma* sp. (Varigated cutworm); *Euxoa* sp. (Army cutworm); and *Agrotis* sp. (Black cutworm):

10 Diptera particularly *Hylemya* sp. (Seedcorn maggot) and *Tetanops* sp. (Sugarbeet root maggot):

Homoptera particularly *Pemphigus* sp. (Sugarbeet root aphid, Cutworm, and White grub) and *Aphis* sp. (Corn root aphid) .

15

The insecticidal coating is particularly useful in accommodating high insecticide loads, as are required to treat typically refractory pests, such as corn root worm, while at the same time preventing unacceptable phytotoxicity due to the increased insecticide load.

20 Most preferred target organisms include in addition to *Diabrotica* sp, *Agrotis ypsilon*; *melanotus cribulosus*; *Hylemya cilicrura*; *Agonoderus lecontei*; *Feltia subgothica* and *Phyllophaga rugosa*.

In other embodiments a plasticizer may be used in the insecticidal solution. Plasticizers
25 are typically used to make the film that is formed by the insecticidal coating layer more flexible, improve adhesion, spreadability and improve the speed during processing. The improved film flexibility is important to minimize chipping, breakage or flaking during handling or sowing processes. Many plasticizers may be used however, most preferred plasticizers include polyethylene glycol, glycerol, butylbenzylphthalate, glycol benzoates and related compounds.
30 The range of the percent of plasticizer in the insecticidal coating layer will be in the range of about 0.1 to about 20%.

In addition to the insecticidal coating layer, the seed may be treated with one or more of the following ingredients: other pesticides including fungicides and herbicides; herbicidal safeners; fertilizers and/or biocontrol agents. These ingredients may be added as a separate layer or alternatively may be added in the insecticidal coating layer.

5

Suitable pesticides include those listed herein and those listed in The Pesticide Manual, 9th Ed., Editor, Charles Worthing, published by the British Crop Protection Council and hereby incorporated by reference.

10 A fungicide may be applied to the seed prior to the coating layer described herein. The application of a fungicide as a dust, slurry or the like is a well known practice in the art and is not considered a coating layer within the meaning of the term used herein. Suitable examples of fungicides include Captan (N-(trichloromethyl)thio-4-cyclohexane-1,2-dicarboximide); Thiram (tetramethylthioperoxydicarbonic diamide; Metalaxyl (methyl N-(2,6-dimethylphenyl)-N-

15 (methoxyacetyl)-DL-alaninate; Fludioxonil (4-(2,2-difluoro-1,3-benzodioxol-4-yl)-1H-pyrrol-3-carbonitrile; and Oxadixyl (N-(2,6-dimethylphenyl)-2-methoxy-N-(2-oxo-3-oxazolidinyl) acetamide. One skilled in the art will be aware of other beneficial fungicides suitable for combating harmful pathogens which are not only a problem for a particular locale where the coated seed is to be grown but also suitable for the protection of seeds in storage before

20 planting.

The amount of fungicide to be added will vary due to the strength of its active ingredient, but in general may range from about 0.001 to about 10% of the weight of the seed and preferably from about 0.01 to 2.0 %. However, for a particular situation the amounts may

25 be greater or less.

Suitable herbicides include those selected from carbamates, thiocarbamates, acetamides, particularly chloroacetamides, triazines, dinitroanilines, glycerol ethers, pyridazinones, uracils, phenoxys, ureas, and benzoic acids and derivatives. Suitable safeners

30 include for example, benzoxazine, benzhydryl derivatives, N,N-diallyl dichloroacetamide, various dihaloacyl, oxazolidinyl and thiazolidinyl compounds, ethanone, naphthalic anhydride compounds, and oxime derivatives.

Suitable biocontrol agents are bacteria of the genera *Rhizobium*, *Bacillus*, *Pseudomonas*, and *Serratia*, fungi of the genera *Trichoderma*, *Glomus*, and *Gliocladium* and mycorrhizal fungi.

- 5 The above compounds are listed as examples and are not intended to be an exhaustive list of compounds that can be used in the insecticidal coating layer or in additional other coating layers.

Conventional means of coating may be used for carrying out the coating of the
10 invention. Additionally, various coating machines are available to one skilled in the art. Three well known techniques include the use of drum coaters, and fluidized bed techniques. Other methods, such as spouted beds may also be useful. The seeds may be presized prior to coating. After coating the seeds are dried and then optionally sized by transfer to a sizing machine. These machines are known in the art for example, a typical machine used when
15 sizing seed corn in the industry.

Film-forming compositions for enveloping coated seeds are well known in the art, and a film overcoating can be optionally applied to the coated seeds of the present invention. The film overcoat protects the coating layers and optionally allows for easy identification of the
20 treated seeds. In general, additives are dissolved or dispersed in a liquid adhesive, usually a polymer into or with which seeds are dipped or sprayed before drying. Alternatively a powder adhesive can be used. Various materials are suitable for overcoating including but not limited to, methyl cellulose, hydroxypropylmethylcellulose, dextrin, gums, waxes, vegetable or paraffin oils; water soluble or water disperse polysaccharides and their derivatives such as alginates,
25 starch, and cellulose; and synthetic polymers such as polyethylene oxide, polyvinyl alcohol and polyvinylpyrrolidone and their copolymers and related polymers and mixtures of these.

Further materials may be added to the overcoat including optionally plasticizers, colorants, brighteners and surface active agents such as, dispersants, emulsifiers and flow
30 agents including for example, calcium stearate, talc and vermiculite. Additionally pesticides, such as fungicides may be added to the film coat, however, it has been observed that

fungicides added initially to the seed gives better results than when added with the overcoat. Fluidized-bed and drum film coating techniques described above can be employed for film coating.

5 The overcoating film is in the range of approximately 0.01% to about 20% of the weight of the seed it is applied to. The preferred range will be about 0.01 % to about 10.0% and a most preferred range will be about 0.01 to 5.0%. A preferred solution will vary depending on the specific active ingredient. A preferred overcoating film may include methyl cellulose, hydroxypropylmethylcellulose, polyvinyl acetate, PEG and mixtures thereof. Additionally,
10 depending on the type of insecticide the overcoat film may have incorporated therein a bird repellant compound. These compounds are known in the art and include for example; anthraquinone, methyl anthranilate, capsaicin and oxygenated tetracyclic triterpenoid compounds (also referred to as cucurbitacins) and disclosed in US Patent 5,292,533. Moreover these compounds may be added to the insecticidal coating layer as well.

15 Depending on the germplasm and crop seed to be coated other benefits may be seen by subjecting the seeds to additional treatments prior to coating such as priming or treatment with growth regulators and biocontrol agents.

20 In seed priming, the seeds are exposed to enough moisture to enhance germination, but not enough to cause premature germination. Examples of priming procedures are known in the art and include drum priming and steep priming.

25 Growth regulators which may be added prior to coating includes auxins, cytokinins, gibberellins, ethylene, abscisic acid, IAA, NAA, IBA, ethephon, acetamide, maleic, hydrazide, diminozide and like compounds. Biological treatment may also be used to enhance seed performance and help in the control of harmful organisms.

30 Seeds Treated with the coating of the invention have the following advantages:
They possess less risk with respect to operator handling and exposure because of reduced dust exposure. Pesticides can be applied in a uniform way and loss of pesticides during transport and handling is prevented. Phytotoxicity is reduced to the seeds and developing seedlings and

therefore there is no adverse effect on germination, seedling emergence and seedling development. Additionally, placement of pesticides, particularly insecticides, closer to the insect soil feeding zone may improve insect control.

- 5 Specific embodiments of the invention are described hereinafter by way of example only.

Example 1 - Seeds treated with an insecticidal coating layer:

- 10 a. Five pounds maize seed pretreated with Captan 400 fungicide and Pro-Ized Red colorant, are coated in an Vector LDCS coating machine. The seeds are warmed for approximately 5 minutes in a rotating pan at approximately 20 r.p.m. with an air flow of 31-32 c.f.m. and an air pressure of 20 p.s.i. A 992 gram insecticide solution comprising 183 grams of 50% chlorpyrifos (the active ingredient in Lorsban™ 50-SL) and 50% premixed inert filler, 47
15 gram Airflex™ 500 (vinyl acetate-ethylene), and 762 grams water is sprayed onto the seed until all the solution is utilized. Inlet air temperature is approximately 50°C and may be periodically adjusted to maintain seed temperature about equal to or less than 35°C or germination may be effected.
- 20 b. Seed is treated as described above however, a 334.0 gram insecticide solution comprising 51.87 grams of 27.9% tefluthrin (the active ingredient in Force™ 30CS), 21.0 grams Vinamul™ 18132 (vinyl acetate-ethylene), 30 grams of Celite™ 266 and 231.0 grams water is applied to the seed.
- 25 c. (1) Seed is treated as described above however, a 900 gram insecticide solution comprising 15.5 grams of 92% tebupirimphos (the active ingredient in Mat™7484), 90 grams of Celite™ 266, 22 grams Airflex™ 500 (vinyl-acetate-ethylene), and 772 grams water is applied to the seed.
- 30 (2) Seed is treated as described in c. (1), except no Celite™ 266 is added to the solution.. A total of 30 grams of calcium carbonate is applied to the seed during the coating process as a drying charge.

- d. Seed is treated as described above however, a 293 gram insecticide solution comprising 11.4 grams of 80% fipronil (the active ingredient in Regent™), 22.6 grams Celite™ 266, 9 grams Airflex™ 500 and 250 grams water is applied to the seed.
- 5 Lorsban™ 50-SL; active ingredient is = o,o-dimethyl 0-(3,5,6 trichloro-2-pyridyl) phosphorothioate, Dow Elanco
- Force™ 30CS; active ingredient is = [1 α , 3 α (Z)]-(\pm)-(2,3,5,6-tetrafluoro-4-methylphenyl)methyl 3-(2-chloro-3,3,3-trifluoro-1-propenyl)-2,2-dimethylcyclo-
- 10 propanecarboxylate, Zeneca, Inc.
- Mat™ 7484; active ingredient = o-[2-(1,1-dimethylethyl)-5-pyrimidinyl-o-ethyl o-(1-methylethyl) phosphorothioate, Bayer
- 15 Regent™ 80WDG; active ingredient = 5-amino-1-(2,6-dichloro-4-trifluoromethylphenyl)-3-cyano-4-trifluoro methane sulphinyl pyrazole, Rhone-Poulenc

Example 2 - Overcoating of coated seeds:

- 20 To each of the seed examples described in Example 1 is added a film overcoat. The same machine and set points are used for the overcoat. The machine is set at an inlet temperature of 50°C which is periodically adjusted to maintain seed temperature about equal to or less than 35°C. A solution comprised of 438 grams water, 2.2 grams Methocel K100M, 0.36 grams PEG 8000, 5.8 grams Pro-Ized red colorant 0.73 grams GP Hytech Calcium
- 25 stearate 5899 and 22.2 grams Airflex is sprayed on the seed for 15 minutes.

Example 3 - Seed Germination and Field Emergence Results:

- 30 Corn hybrid seed germination is tested under testing rules of Association of Official Seed Analysts (AOSA). Both paper towel and soil are used for separate germination tests. In addition, seedling shoots and roots were separated after 7 to 10 days of germination. Dry weight of shoots and roots is recorded to indicate seedling growth. For field emergence test,

corn hybrid seed is planted in Iowa, Minnesota, Wisconsin, Nebraska, Illinois, and Ohio in a split plot design with two rows per treatment and four replications. Emergence counts are taken every other day beginning with the first day plants emerge for each treatment and continue until 21 days after emergence. The % emergence is determined for all locations for each treatment and is exhibited in Table 1. This Table demonstrates that seed coating does not adversely affect seedling emergence. The treatments are described as follows:

Treatment 1(a): Seed is treated as described in Example 1(a);

10 Treatment 1(b): Seed is treated with chlorpyrifos at the same rate as described in Example 1(a), except that no binder (Airflex™ 500) is used.

Treatment 2: Seed is treated as described in Example 1(b);

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Treatment 3: Seed is treated as described in Example 1(c)(1);

Treatment 4: Seed is treated as described in Example 1(d);

20 Treatment 5: Seed is treated with Captan 400 fungicide;

Treatment 6: Seed is treated as described in Example 2;

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TABLE 1

Treatment	% Germination			Shoots(mg)	Roots(mg)	% Corn Emergence
	Paper	Soil				
		4 Days	7 Days			
1(a)	99	95	95	10.2	12.55	88
1(b)		70	80*			
2	98			-	-	81
3	98			13.5	11.6	87
4	99			13.1	12.95	86
5	99			-	-	87
6	99	95	97	11.67	13.14	88

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* Seedlings are not uniform and grow very slowly.

Example 4 - Effect on Phytotoxicity of Using Filler In the Coating

A high resolution saturated cold test was used to test the effect of filler on the reduction of phytotoxicity. In this example seeds were germinated at 10°C with low oxygen, and a water saturated condition for 5 days, and then transferred to 25°C to finish the germination process. The % saturated cold germination and % field emergence is exhibited in Table 2.

TABLE 2

Hybrid Seed	Treatment	% Sat. Cold Germ	% Corn Emergence
N4242	C (1)	72	88
N4242	C (2)	66	84
N7590	C (1)	71	
N7590	C (2)	54	

The results indicate that the formulation containing a filler (C(1)) provides an improvement in % germination and emergence.

Example 5 - Corn rootworm efficacy studies:

Hybrid corn seeds with and without an insecticidal coating are planted in Nebraska, Iowa, Minnesota, Wisconsin and Illinois to determine the efficacy of various coated seed treatments in standard tests. All seeds contain a Captan pretreatment whether or not a coating is applied. Additionally, an overcoat layer is applied to all coated seed which comprises the coating described in Example 2. Banded granular insecticides are applied at the manufactures label rates using granular insecticide applicators. Plot size for each treatment is two ten foot rows spaced 30 inches apart with four replications of each treatment at each location. Non-diapausing 1st instar Western Corn Rootworm larvae are side dressed on both sides of the corn plants in the row at a rate of 800 eggs per foot of row. Application of the insect larvae is made when the corn seedlings are approximately at the two leaf stage. The placement of the larvae is approximately two inches to the side of the corn plants and two inches below the soil surface. Plots are maintained with normal cultural practices until the corn plants reach the

flowering stage. At late brown silk stage, 10 sequential roots from each plot are obtained. The corn stalks are cut at approximately 12 inches above the soil line. Each corn root is washed and after washing the roots are rated on a scale of 1 - 6 using the Iowa Root Rating Method developed at Iowa State University (Hills and Peters, 1971) and used commonly throughout the corn belt. In the rating, 1 = no damage or only a few minor feeding scars; 2 = feeding scars evident but no roots eaten off to within 1 1/2 inch of the plant; 3 = several roots eaten off to within 1 1/2 inch of the plant but never the equivalent of an entire node of roots is destroyed; 4 = one root node completely destroyed; 5 = two root nodes completely destroyed and 6 = three or more root nodes destroyed. A destroyed root is defined as a root that has been pruned to within 1 1/2 inch of the base. Pruned roots do not have to originate from a single node, but all pruned roots must equal the equivalent of a full node to count as a destroyed node. The mean root rating results are reported in Table 2 below. In addition, a growth chamber and a greenhouse bioassay was used to determine the effect of insecticidal seed coating on corn rootworm control. For growth chamber assay, corn seed was planted in a 16 oz cup with garden Jiffymix, and allowed to grow for two weeks. Twenty neonate corn rootworm larvae are placed in the Jiffymix near corn roots and allowed to feed for approximately 2 weeks. The larvae are then screened out and the % mortality of the larvae is determined. Each corn root is washed and after washing the roots are rated on a scale of 1 - 6 using the Iowa Root Rating Method developed at Iowa State University (Hills and Peters, 1971). Some of the results are indicated below in Table 3. The greenhouse assay use 2 gallon pot with 20 mesh corn field soil. Forty corn rootworm larvae are placed in each pot when corn plant was at 4 to 5-leaf stage, and allowed to feed for 2 to 3 weeks. Each corn root is washed and after washing the roots are rated on a scale of 1 - 6 using the Iowa Root Rating Method developed at Iowa State University (Hills and Peters, 1971). Some of the results are indicated below in Table 3.

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30

TABLE 3 - Corn Rootworm Field Efficacy Results

<u>Treatment</u>	<u>Root Rating</u>
Captan only	4.7
Overcoating only	4.5
Example 1(a)	3.43
Example 1(b)	3.0
Example 1(c)(2)	3.2
Example 1(d)	3.55
Lorsban™ 15 G soil T-band*	3.0
Aztec™ 2.1G soil T-band	2.8
Force™ 1.5G soil T-band	2.6

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Lorsban 15 G = 15% 0,0-diethyl-0-(3,5,6-trichloro-2-pyridyl)phosphorothioate, DowElanco

10 Aztec 2.1 G = 2% 0-[2-(1,1-dimethylethyl)-5-pyrimidinyl-o-ethyl o -(1-methylethyl) phosphorothioate and 0.1% cyano(4-fluoro-3-phenoxyphenyl)methyl 3-(2,2-dichloroethyl)-2,2-dimethylcyclopropanecarboxylate, Bayer

Force 1.5G = 1.5% [1 α , 3 α (Z)]-(\pm)-(2,3,5,6-tetrafluoro-4-methylphenyl)methyl 3-(2-chloro-3,3,3-trifluoro-1-propenyl)-2,2-dimethylcyclopropanecarboxylate, Zeneca, Inc.

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20

* Manufacturer's label rates and application instructions are followed.

TABLE 4 - Corn Rootworm Bioassay Results

Treatment	Growth Chamber		Greenhouse
	% Mortality	Root Rating	Root Rating
Captan only	27	5.57	4.02
Example 1(a)	87	3.55	3.15
Example 1(c)	96	1.44	2.15
Example 1(d)	80	3.75	--

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Example 6 - Corn rootworm efficacy studies:

10 Maize seed is treated as described in Examples 1 and 2. A combination of methyl cellulose and polyvinyl alcohol is used as a binder and Celite 266 is used as filler for insecticide solution, which contains the compound of formula II as the insecticide. Bioassay methods are as described in Example 4. Root rating was based on the Iowa State 1-6 scale.

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TABLE 5 - Corn Rootworm Bioassay Results

	Growth Chamber (16 oz cup)	Greenhouse (2 gal pot)
Treatment (mg/seed)	Root Rating	Root Rating
Control	5.69	4.54
Tefluthrin 50% (1.0)	2.30	3.52
Formula II (0.01)	5.30	
(0.03)	4.30	
(0.3)	2.70	
(0.6)	2.10	2.73
(0.8)	2.30	
(1.0)	2.10	
(1.2)		3.08
(1.5)		2.96
(2.0)		2.90
Raze* /Formula II (0.01)	2.48	2.63
Raze*/Formula II (0.3)	1.87	2.34

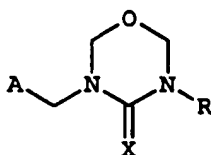
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* RazeTM is a tefluthrin seed treatment formulation (0.13 mg/seed) marketed by Wilber Ellis for wireworm control. RazeTM seed treatment alone did not show any significant activity against CRW.

WHAT IS CLAIMED IS:

1. An insecticidal coating for a seed comprising:
 - 5 a) one or more binders selected from the group consisting of polymers and copolymers of polyvinyl acetate, methyl cellulose, polyvinyl alcohol, vinylidene chloride, acrylic, cellulose, polyvinylpyrrolidone and polysaccharide;
 - b) an insecticidally effective amount of an insecticide for the control of
10 Diabrotica or its larvae; and
 - c) one or more fillerswherein the binder forms a matrix for the insecticide and filler and is present in an amount effective to prevent or reduce the phytotoxic effect on the seed caused by the insecticide.
15
2. The coating of claim 1 further comprising a film overcoat.
3. The coating of claim 1 wherein the seed is selected from the group consisting of soybean, sugarbeet, corn, sunflower, alfalfa, sorghum, rapeseed and tobacco.
20
4. The coating of claim 3 wherein said seed is corn.
5. The coating of claim 1 wherein the filler is an absorbent filler selected from the group consisting of diatomaceous earth, perlite, silica, calcium carbonates, and mixtures
25 thereof.
6. Seed coated with the coating according to claim 1.
7. ~~The~~ The coating of claim 1 wherein the insecticide is imidacloprid, an imidacloprid
30 analog, an organophosphate, phenylpyrazole or pyrethroid insecticide, or combinations thereof.

8. The coating of claim 7 wherein the insecticide is a compound of the general formula (I)

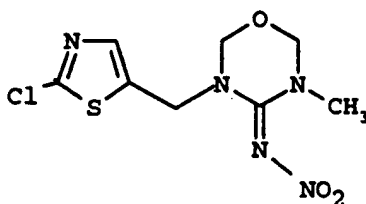


(I),

in which

- A is an unsubstituted or mono- to tetrasubstituted, aromatic or non-aromatic, monocyclic or bicyclic heterocyclic radical, where one to two of the substituents of A can be selected from the group consisting of halo-C₁-C₃alkyl, cyclopropyl, halocyclopropyl, C₂-C₃alkenyl, C₂-C₃alkynyl, halo-C₂-C₃alkenyl, halo-C₂-C₃alkynyl, halo-C₁-C₃alkoxy, C₁-C₃alkylthio, halo-C₁-C₃alkylthio, allyloxy, propargyloxy, allythio, propargylthio, haloallyloxy, haloallythio, cyano and nitro, and one to four of the substituents of A can be selected from the group consisting of C₁-C₃alkyl, C₁-C₃alkoxy and halogen;
- R is hydrogen, C₁-C₆alkyl, phenyl-C₁-C₄alkyl, C₃-C₆cycloalkyl, C₂-C₆alkenyl or C₂-C₆alkynyl; and X is N-NO₂ or N-CN, or, if appropriate, a tautomer thereof, in each case in free form or in salt form, terbufos, chlorpyrifos, fipronil, tefluthrin, chloroethoxyfos or tebupirimfos, or combinations thereof.

9. The coating of claim 8 wherein the insecticide is the compound of formula II



(II)

and optionally tefluthrin.

10. The coated seed of claim 6 further comprising a fungicide.
11. The coating of claim 1 further comprising a plasticizer.
12. The coating of claim 1 further comprising a bird repellent compound.
13. The coating of claim 1 wherein the filler comprises a mixture of diatomaceous earth and silica.
14. The coating of claim 1 wherein the binder is in the range of 0.01 to 15% of the weight of the seed, the filler is in the range of 0.01 to 50% of the weight of the seed, and the insecticide is in the range of 0.005 to 50% of the weight of the seed.
15. An insecticidal coating for a corn seed comprising a binder in an amount from about 0.01 to about 15% of the weight of the seed wherein said binder is a vinyl acetate-ethylene copolymer or polymer or copolymer of vinylidene chloride, a filler in an amount from about 0.01 to about 50% of the weight of the seed wherein said filler is diatomaceous earth and amorphous silica, and an insecticidally effective amount of a compound effective against corn root worm or its larvae selected from compounds of the group consisting of the compounds of formula II, imidacloprid, tefluthrin, and combinations thereof, and in the range from about 0.005 to about 50% of the weight of the seed.
16. A method of controlling insect pests from damaging a crop plant comprising applying to a seed an insecticidal coating according to claim 1.
17. A method of controlling insect pests from damaging a crop plant comprising applying to a seed an insecticidal coating according to claim 7.
18. The method according to claim 16 wherein the seed is corn.
19. The method according to claim 16 wherein the insect is a Diabrotica insect.

20. The method according to claim 16 wherein the insect is corn root worm. —

21. A method of preparing coated seeds comprising:

5 a) mixing a binder selected from vinyl acetate-ethylene copolymers or polymers or co-polymers of vinylidene chloride or mixtures thereof, and a filler selected from diatomaceous earth, perlite, silica, calcium carbonates and mixtures thereof, with a terbufos, chlorpyrifos, tefluthrin, fipronil or tebupirimfos insecticide;

10 b) applying the mixture to a seed;

c) allowing the mixture to dry on said seed; and

d) applying a film overcoating to the seed.

15

22. A seed coated according to the method of claim 21.

23. A method of protecting emerging seedlings of a crop plant from one or more insect organisms in the seed growing soil environment which comprises applying to the seeds
20 of the crop plant an insecticidally effective amount of a coating according to claim 1.

24. The method of claim 23 wherein the crop plant is corn.

25. The method of claim 23 wherein the insect is a Diabrotica insect.

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26. The method of claim 25 wherein the insect is corn root worm.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/EP 98/08171

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 A01N25/10 A01N25/32 A01C1/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 A01N A01C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 383 391 A (THOMAS NORMAN W ET AL) 17 May 1983 (1983-05-17) column 1, line 8 - line 26 column 1, line 66 - column 2, line 13 column 2, line 35 - line 60 column 4, line 6 - line 35 column 4, line 55 - column 5, line 5 column 5, line 60 - column 6, line 17; claims 1,10,14	1,3-6, 10,13, 14,16, 18,23-26
X	GB 2 040 684 A (FMC CORP) 3 September 1980 (1980-09-03)	1,3,4,6, 14,16, 18-20, 23-26
Y	page 1, line 23 - line 60; examples 1-9,11; tables 1,5	2,7-12, 17,21,22

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☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 98/08171

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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Y	EP 0 580 553 A (CIBA GEIGY AG) 26 January 1994 (1994-01-26) page 2, line 1 - line 27 page 7, line 47 - line 52 page 13, line 32 - line 34 page 15, line 46 - line 53 table 1, compound no. 1.3 claims 26, 27; example B5 ---	7-9, 17
Y	EP 0 010 630 A (SAREA AG) 14 May 1980 (1980-05-14) page 2, line 13 - line 21 page 3, line 4 - line 16; example 4 ---	10, 12
A	EP 0 091 213 A (FMC CORP) 12 October 1983 (1983-10-12) page 1 - page 4, paragraph 2 page 5, last paragraph - page 6, paragraph 1 page 7, last paragraph -----	1-26

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 98/08171

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